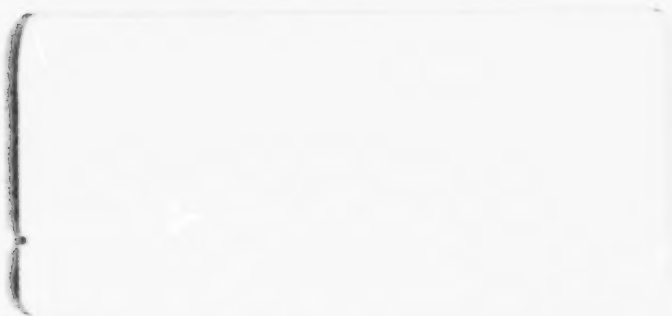


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Sherritt Research Centre

COMPACTING CHARACTERISTICS OF
RAPIDLY SOLIDIFIED PARTICULATES

FINAL REPORT

March 1986

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**Compacting Characteristics of
Rapidly Solidified Particulates**

Final Report

by

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Work on this project was conducted under the auspices of the Canada Centre for Mineral and Energy Technology, Energy, Mines and Resources and The Industrial Materials Research Institute, National Research Council of Canada.

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SUMMARY

A melt extraction apparatus was constructed and proved able to produce small quantities of fibers having uniform length. Fiber length could be changed by altering the notch to notch spacing on the 151 mm diameter copper extraction wheel. A useful operating speed range for the wheel was established but run time was limited to about 2 min to prevent overheating and damage to the wheel. Modifications were recommended to allow production of larger batches.

The working metal was pure nickel and cross-section metallography showed equiaxed or columnar grain structures, the latter being nucleated at the wheel contact surface. To what extent the melt extraction process could create non-equilibrium phase compositions or microstructures could be determined using a suitable multi-phase alloy.

Fiber characterization included oxygen analysis which showed the fibers to have a thicker surface oxide than atomized powders, but not considered high enough to adversely affect a wrought microstructure. Comparative hardness tests showed that the fibers ranked between hard hydrometallurgical and soft atomized particulates although closer to the latter. MPIF standard tests were conducted for transverse rupture and tensile strengths and showed that compacts of larger fibers were stronger.

Roll compacting studies showed that fibers blended with hydrometallurgical powder made more flexible strip than when blended with atomized powder. Fibers tended to align in the rolling plane, suggesting a possible reinforcement role or a way to make interesting anisotropic composites. It was concluded that larger quantities of fiber and an extensive experimental program would be necessary to establish a practical roll compacting process and product using melt extracted nickel fibers alone.

SOMMAIRE

Un appareil servant à l'extraction d'un matériau à l'état de fusion a été construit. Il s'est avéré efficace pour produire de petites quantités de fibres ayant une longueur uniforme. La longueur de la fibre peut être modifiée en changeant la distance entre les coches sur la roue d'extraction de 151 mm en cuivre. Un intervalle de vitesses d'opération pour la roue a été établi; par contre le temps d'opération a été limité à deux minutes afin de prévenir le surchauffement et l'endommagement de la roue. Certaines modifications ont été recommandées afin d'être capable de produire de plus grandes quantités de matériaux.

Le métal utilisé était du nickel pur et une coupe métallographique transversale a montré une structure de grain soit équiaxe ou soit en colonne, cette dernière étant nucléée à la surface de contact avec la roue. Pour déterminer jusqu'à quel point le procédé d'extraction peut créer des microstructures ou des phases non-équilibrées on pourrait utiliser un alliage à plusieurs phases approprié.

La caractérisation de la fibre a inclus l'analyse de l'oxygène qui a indiquée une surface oxydée plus épaisse que dans le cas des poudres atomisées, mais pas assez pour causer des effets adverses sur la microstructure forgée. Des tests comparatifs de dureté ont indiqué que les fibres se situaient parmi les particules hydrométallurgiques à haute dureté et les particules atomisées à basse dureté, quoique plus près de ces dernières. Des tests standards de rupture transversale et de résistance à la traction du MPIF ont montré que les échantillons préparés à partir de fibres plus grandes étaient plus forts.

Des études sur le compactage de fibres par laminage à froid ont montré que les fibres mélangées avec de la poudre hydrométallurgique produisent des bandes plus flexibles que dans le cas de celles mélangées avec de la poudre atomisée. Les fibres tendent à s'aligner parallèlement à la surface de la pièce laminée suggérant un rôle de renforcement possible ou une façon de faire des composés anisotropes intéressants. Il a été conclu que de plus grandes quantités de fibres et un vaste programme expérimental seraient nécessaires pour établir un procédé pratique de laminage et un produit utilisant seulement des fibres de nickel extraites à partir du nickel en fusion.

1.0 INTRODUCTION

Rapid solidification (RST) is a new technology that has attracted world-wide interest and is being pursued actively, particularly in Japan and the U.S. In Canada, a number of groups are studying the opportunities for RST, and universities and other institutions such as EMR/CANMET have begun research programs. RST promises potential improvements in a number of material properties in almost all known alloy systems.

With the exception of the so-called metal glasses that are prepared in continuous ribbons by the melt spinning techniques, most of the material property studies are being done on small individual alloy particulates that have no commercial use per se. A major requirement for utilizing RST material properties is that methods must be developed for consolidating the RST particulates into useful commercial shapes and wrought products. Continuous direct powder roll compaction, a commercial process that was developed by Sherritt Gordon in Canada for consolidating nickel powder into nickel strip(1), has some of the process characteristics that are required for successful conversion of the RST particulates into a useful wrought product.

Sherritt Gordon Mines Limited was engaged by the Federal Department of Supply and Services (DSS) to conduct a research program under the auspices of the Canada Centre for Mineral and Energy Technology, Energy, Mines and Resources, (CANMET) and the Industrial Materials Research Institute, National Research Council of Canada. The technical guidance for the project was provided by the Scientific Authority at CANMET whilst the contractual guidance came from the Science Procurement Officer at DSS. The effective commencement date was January 1, 1985.

The objective of the program was to conduct exploratory experiments to assess the feasibility of consolidating RST (rapid solidification technology) particulates by roll compaction. The technical program that has been completed, and is now to be discussed, comprised the design and construction of a melt extraction unit, the production of particulates using various combinations of operating parameters, the characterization of particulates using standard metallurgical techniques, and static and roll compactability tests.

Appendix 1 shows the Contract Plan and Report Form. This details the chronological progression of technical tasks and the technical discussions to follow in sections 2.0 and 3.0 will follow that format.

A series of monthly progress reports was issued during the course of the contract which contained comprehensive details of the work done and included tables, diagrams and photographs. In order that the Final Report be complete in itself, much of the data already presented is reproduced and consolidated here.

2.0 MELT EXTRACTION

2.1 Apparatus Design and Construction

The first task in the project was the design of, and materials procurement for, a melt extraction unit. A simple schematic of the melt extraction process is shown in Figure 1. Essentially the design comprises a variable speed rotating copper wheel that interacts with the surface of a pool of molten metal. As discussed in the proposal, the working metal in the program was pure nickel, chosen because of our know-how in roll compacting a variety of nickel powders.

It was decided from the outset that the molten metal would be supplied from an existing laboratory induction melting furnace which has a 6.5 kg capacity (Ni). The interior diameter of the furnace was not large enough however to admit a melt extraction wheel. The first idea therefore was to pour the molten metal into a preheated, but not self heating, tundish then either raise the tundish against the spinning wheel or lower the wheel onto the molten metal surface.

Tests of the preheated, but not self heating, tundish approach were conducted using the best refractory technology that had been established in our production atomizing plant. However, molten nickel holding tests indicated that the metal surface froze after only about 90 s, so this approach was clearly not viable. The next logical step, of constructing a self heating (e.g. induction wound) tundish was put off pending the result of a different approach, due to projected costs and time delay.

The alternative approach was to modify the pouring lip of our crucible induction furnace so that when the furnace was tilted a pool of molten nickel would form in the lip extension, and the geometry of the system would allow approach and contact by the spinning extraction wheel. Tests indicated that this approach would work, and so design of the extraction unit itself proceeded on this assumption.

Figure 2 shows a simplified cutaway drawing of the melt extraction assembly that was built. It can be seen that the spinning wheel is part

of a sub-assembly that can be moved in a vertical direction using precision screw jacks (shown) operated by a handwheel (not shown). Not shown is a tachometer attachment for measuring disc rotation speed. The assembly runs on tracks so that it can be accurately located over the fixed position furnace.

Figure 3 shows a photograph of the completed unit in operating position, although not with a melt in progress. The copper melt extraction wheel can be seen in position over the lip of the tilted induction furnace. Not visible is a contra-rotating wire brush wheel on top of the copper disc which wipes off any nickel that is carried around the circumference. In addition to ON/OFF power switches, the control panel has a speed control which allows a disc drive shaft speed range of 0 to 180 rpm. For the extraction wheel diameter of 151 mm, this translates to a wheel surface speed range of 0 to 14.2 m/s. The tachometer is a non contact type with a light beam sensing a reflector on the drive shaft. The screw jack handwheel can move the disc assembly through 0.635 mm vertically for each full turn. It should be noted that the fiber shield and associated, removable, collecting tray were added part way through the program. Prior to that, the extracted fibers were collected off the floor. Not completely shown in Figure 3 is a fume extraction system that was installed for the program to protect the unit operator during meltdown and extraction.

2.2 Operating Variable Studies

2.2.1 Commissioning

The unit was commissioned using a smooth, unnotched copper wheel with dimensions shown in Figure 4. The combination of apex angle and radius chosen was somewhat arbitrary, having no experience to go on, but with the intention that it would allow easy separation of the fibers and, if the melt froze only on the radius, section widths of ~1 mm.

Prior to this first, and all subsequent runs, the wheel periphery was given a 600 grit finish (100 to 400 nm). A full charge of nickel (6.5 kg) was preheated to 1670°C (i.e. 227°C superheat), this temperature was shown in later runs to be about the maximum practical pouring temperature to avoid furnace refractory damage. The furnace was tipped to bring molten metal into the extended furnace spout, the extraction frame was then wheeled over the furnace and the spinning wheel lowered, using the screw jack hand wheel, to make contact with the metal in the spout. About 200 g of fibers up to 100 mm long were produced in 1 to 2

min of contact time before the spout froze up. All equipment worked as planned and it appeared that sufficient material could be produced to meet the program objectives. Commissioning was therefore considered to be complete and the above operating procedure used in all subsequent runs.

2.2.2 Operating Variables and Fiber Characterization

The first study conducted was a four run series with a smooth unnotched wheel and rotation speed as the variable. Table 1 summarizes the operating parameters and recoveries and the following observations were made.

- At higher wheel speeds the system was harder to control. Fibers tended to be thrown higher and even carried around the wheel before being wiped off by the contra-rotating wire brush. The spout metal cooled more quickly and limited the useful contact time. The wheel heated up more and in C4 there appeared to be some incipient copper melting on the wheel and definite oxidation and roughening. At the two lower speeds the wheel was unscathed. Damage at the higher speeds might be due to the shorter time available for cooling between melt contacts.
- At all speeds the fibers had the same general appearance, i.e. smooth, concave on the wheel side and rough (like a weld bead), convex on the outer side. The outer surface and edge were smoother at higher speeds. All subsequent runs had the same general appearance.

In the second series of runs the same wheel design was used but this time with 18 equally spaced notches around the circumference, as shown in Figure 5. Note the direction of rotation, and that the notch to notch, centre to centre spacing was 26.4 mm and fiber lengths were 22 mm. Table 2 summarizes these runs. Figure 6 shows the external shapes of C6 and C7 fibers. The former are more massive and have more flash, reflecting the operator's observation that at lower wheel speeds it seems harder to take a 'fine cut' at the melt surface. Figure 6 also shows that the notch to notch separation was consistently reproduced in the fiber length in each run; this was also the case in later runs with much shorter notch to notch spacings. The shovel end forms first and a clean break is made at the perpendicular face of the next notch. In

later studies making shorter fibers the same general surface appearance was evident although the 'shovel' made up a larger proportion of a fiber's mass.

Figure 7 shows a range of transverse section shapes and sizes typical of all the melt extraction runs in the program. Section widths generally varied in the range 0.2 to 1.2 mm and maximum section thicknesses varied in the range 50 to 250 μm . To produce sections of a more consistent thickness a constant speed motorized vertical drive would be desirable in place of the present manual movement.

The runs summarized in Table 2, and all subsequent runs, generally had fibers with similar grain structures. In some cases large columnar grains, nucleated at the the inner (i.e. wheel periphery) surface filled the whole section, as shown in Figure 8A. The presence of columnar grains however does not necessarily conflict with the definition of a 'rapid solidification process. Nucleation at the contact surface can take place in a time interval between liquid metal extraction and solidification that meets rapid solidification criteria. A cooling rate gradient through the thickness of the fiber whilst still in wheel contact would promote columnar grain growth. Columnar structures have been reported in the melt extraction of Ti alloys (2) and the melt spinning of Ni-Mo alloys (3) and Al (4). It has also been observed in Cu alloys that lower melt superheats promote more uniform through section nucleation and hence more equiaxed structures and that more equiaxed structures would result from faster separation from the chill surface (5). The latter might be an explanation for the difference between Figures 8A and 8B.

Following the runs described in Table 2, two attempts were made with a smooth, unnotched wheel having a 60° apex angle, dimensions otherwise were the same as shown in Figure 4. The finer apex angle was chosen in an attempt to smooth the edges of the fibers by reducing freezing on the flat surface of the apex away from the radius section. Table 3 shows that the first run (C11) was short and the second run aborted, both due to furnace power supply problems. This was a frequent problem throughout the rest of the program, despite many efforts to solve it including replacement of some power supply components and lastly, interruption of all likely inductive current loops using ceramic bridges. Following the last named alteration, performance was better although one power trip still occurred. Part at least of the problem may have been caused by use of the furnace in an inclined position with consequent uneven inductive coupling between the furnace coils and the

melt. The fibers produced in C11 were similar in external appearance and cross section to those produced by the 90° apex angle smooth wheel. In runs C11, 13, 15 and 16 the fibers were collected in a water tray placed on the floor. This 'water quench' however had no significant effect on microstructure or external appearance.

Subsequent to C12 a wheel having 54 notches, with centre to centre spacings of 8.8 mm was tried. Dimensions otherwise were the same as shown in Figure 5. Fiber length was 4 mm and Table 3 summarizes the run parameters. A key observation regarding the product of this series of runs was that the short fibers flowed freely compared to the longer fibers produced with the 18 notch wheel which formed tangles. A free flowing product is necessary for any material to be used as feed to a roll compacting mill.

Prior to the next series of runs, summarized in Table 4, an enclosed fiber guide chute and detachable fiber collection tray were installed. The additional 54 notch wheel runs were intended to make more material for compactability tests. In run C21 the fibers were collected in water with fibers being intercepted earlier (i.e. hotter) in their trajectory than in the earlier water quench experiments. Little microstructure refinement was observed however, with many columnar grains still evident.

Also listed in Table 4 is a run using an 18 notch wheel with a shallower notch design (Figure 9). These shallower notches (2 mm deep versus previous 3 mm) also gave clean breaks and uniform fiber lengths but, as anticipated, the shovel ends were smaller.

Prior to the next series of runs, summarized in Table 5, a shield/collecting tray combination was installed between the wheel and the furnace and above the wheel axis, to catch backward flying fibers not caught by the wiper brush. These generally amount to a few percent of the main catch tray recovery. It was also felt that back flying fibers falling on the furnace power supply cable junctions may have contributed to power instability.

Table 5 shows that runs C22, 23 and 24 were made using a 72 notch wheel, dimensions otherwise as shown in Figure 9. Notch to notch, centre to centre, spacing was 6.6 mm, giving fibers about 2 mm long. In an effort to make still smaller fibers, a 120 notch wheel with a 60° apex angle and necessarily shallower notches was made (Figure 10) and successfully tested in runs C25, 26 and 27 (Table 5). This wheel

produced fibers about 1 mm long. With both these wheels much of the mass of each fiber derived from the section that freezes on the notch inset (shovel) rather than on the periphery between notches. It was concluded from the Table 5 series that insufficient quantities of 120 notch wheel fibers could be made for roll compacting studies using the present apparatus.

After run C27 the 72 notch wheel was put back in and three more runs completed to produce extra material for compactability tests (Table 6).

2.3 Comparison With Commercial Particulates

Figures 11, 12 and 13 compare the external shapes of Sherritt's melt extracted fibers with those of particulates made by other companies. It is seen that the Transmet melt extracted aluminum fibers are similar in size to Sherritt fibers although without the shovel end. The Ribtec pictures show that straight or hooked end melt extracted fibers can be made by changes in notch design.

Figures 14 and 15 show that the Transmet and Ribtec particulates generally have more uniform microstructures than Sherritt fibers.

3.0 COMPACTABILITY STUDIES

The objective of these studies was to compare and to contrast the roll compacting characteristics of atomized (MA), hydrometallurgical (BRS) and melt extracted (ME) nickel particles.

3.1 Static Tests

A number of preliminary tests were conducted on both loose and pressed particulates to assess their likely utility in roll compacting.

Surface oxide content can be important both in relation to interparticle friction effects and to wrought microstructure cleanliness. Table 7 shows oxygen contents determined by a hydrogen loss technique. Whereas the given oxygen contents should accurately represent the surface oxides of the ME and MA particles, the oxygen content of the BRS powder may also represent occluded salts such as ammonium sulphate.

All else being equal, oxygen content would be expected to increase with specific surface area. However although MA powder has a larger specific surface area than any of the ME particle batches, Table 7 shows that it has a significantly lower oxygen content, i.e. thinner surface oxide layer. The oxygen contents of the ME fibers however are not out of line with those of other water atomized powders such as cobalt alloys, which have been roll compacted successfully and then worked to give clean microstructures with good properties.

An important physical property influencing compacting behavior is particle hardness, i.e. its ability to deform plastically under stress. Earlier studies at Sherritt have shown that for a given stress, significantly higher compacted densities are obtained from water atomized nickel powder (soft) compared to hydrometallurgical nickel powder (hard)(6). Table 8 shows that the melt extracted fibers are harder than atomized particles but softer than hydrometallurgical particles.

One measure of compactability used at Sherritt as a quality control tool is an MPIF transverse rupture test. In this test a rectangular compact is supported near each end and a top load applied in the centre. The transverse rupture strength (TRS) is calculated knowing the load to rupture and the physical dimensions of the compact. Table 9 shows TRS values for melt extracted, water atomized and hydrometallurgical nickel particulates. Three groups of melt extracted fibers were tested, with compacts of the largest fibers (C19, 20 and 21) being stronger than the middle sized fibers (C22, 23 and 24) which in turn were stronger than the smallest fibers (C25, 26 and 27). For a given compacted density, the smallest fibers had a TRS similar to atomized powder. Note the low densities of the compacted hard hydrometallurgical powders.

Another measure of compactability is an MPIF tensile test for which samples are made using a dogbone shaped die. Table 10 shows that in this test, atomized powder and the smallest fibers were weaker than the other two fiber groups although this would be partly due to the higher compacted density of the latter.

Apart from the poor flow properties of the longer fibers it was concluded from these tests that melt extracted fibers generally were suitable for roll compacting.

3.2 Roll Compaction

A series of roll compacting tests was conducted on nickel powders (controls) and nickel powder/melt extracted nickel fiber blends. Table 11 shows roll compacting conditions and dimensions of the product and also shows that all fiber batches used were made with the 72 notch wheel. The small laboratory roll compacting mill was used which had rolls of 150 mm diameter rotating at 9.9 rpm. The mill had no load meter. For each run, a 200 g powder/fiber blend was mixed with 0.5% H₂O then fed into a 0.51 mm roll gap. It is seen that water atomized powder makes thicker, denser strip than hydrometallurgical powder (tests 1, 3 and 6), a fact observed in Sherritt's production roll compacting operation (560 mm roll diameters). The addition of melt extracted fibers to either of the above powders increases strip thickness and density (test 2, 4, 5 7 and 8). The increased thickness is attributed to greater particle/particle and roll/particle friction which enhances the feed rate into the roll gap whilst the increased density is attributed to the ability of the fibers, like atomized particles, to deform and conform (6). Generally only fragments of green strip were produced and production became more difficult as fiber volume loading increased, with strip from blends with the atomized powder being particularly stiff, brittle and difficult to handle. Flexibility is an important factor at Sherritt where roll compacted strip has to be coiled, transported then uncoiled into a sintering furnace. For atomized powders a 25% fiber addition seemed about the limit, whilst higher loadings would likely be possible with the hydrometallurgical powder.

Figures 16 and 17 show typical metallographic cross sections of roll compacted strip from some of the tests. Note how readily the fibers align parallel to the plane of rolling, suggesting that roll compacting of powder/melt extracted fiber blends might be a route to fiber reinforced materials and other composite structures. The microstructural differences between the hydrometallurgical and atomized powder blends are clearly shown. Note especially how the softer fibers and atomized particles deform and mold against each other during roll compaction (e.g. test 5) whilst the harder hydrometallurgical particles interlock with each other but do not deform or mold with the fibers (e.g. test 8).

4.0 CONTRACT PLAN

Appendix 1 shows the Contract Plan and Report Form. Design and Procurement took longer than originally planned due to the need for furnace experiments to be completed before the design could be fixed,

and because some materials e.g. copper for the wheel, took longer to deliver than expected. The Construction task was stretched out over a longer time period than Plan, reflecting an overlap with the Design and Procurement phase, i.e. building as parts became available. The Operating Variable Study started close to plan but lasted longer due to a decision to try additional multi-notched extraction wheel configurations. The Characterization task ran longer than Plan due to a decision to make oxygen content measurements. Compactability studies ran a month later than Plan to accommodate additional product made during an extension of the Operating Variable study. Drafting of the Final Report started late to accommodate the extensions in the Characterization and Operating Variable Studies.

Costs generally ran to Plan for most of the Tasks. However the Operating Variable Study cost more than planned due to the scope extension (additional extraction wheels), whilst the Compactability Studies were underspent due to the limited amount of fiber product available and physical limitations on the product fiber/nickel powder blend combinations that could be tested in roll compaction. Total manhours for the program were according to Plan.

5.0 CONCLUSIONS AND RECOMMENDATIONS

The program demonstrated that nickel fibers of controlled length could be produced using a simple melt extraction apparatus. However in any future work it is recommended that changes be made to allow longer run times and larger volumes of fiber product. These changes should include a fixed induction furnace with an oval plan section large enough to admit the extraction wheel. Use of a furnace in the upright position should improve power stability. Water cooling of the wheel would allow longer metal contact times and multiple rims on the extraction wheel would also increase product output. It would be desirable to have better control of fiber thickness, especially in any program where alloy phase and microstructure variations were planned by virtue of cooling rate, which is sensitive to section thickness. Motorized control of the vertical movement of the extraction wheel is thus recommended.

Four fiber lengths were produced using 18, 54, 72 and 120 notch wheels respectively. The lengths were 22, 4, 2 and 1 mm and all except the first flowed freely like a powder, an important consideration when filling dies or roll gaps for compacting studies. It would be useful to determine the maximum fiber length that still has good bulk handling properties, i.e. to investigate the 4 to 22 mm range.

The frequent occurrence of columnar grains in the nickel fiber microstructures is an indication of cooling rate gradients and suggests that perhaps only part of the structure could truly be termed 'rapidly solidified'. To what extent this melt extraction process could create non-equilibrium phase compositions or microstructures could be determined using a multi-phase alloy.

Physical tests on extracted fibers consolidated by die pressing showed that they were capable of making strong but rather inflexible compacts. Roll compacting studies were limited to blends of fibers and powders and resulted in some interesting microstructures. In particular the alignment of the fibers in the rolling plane suggests that roll compacting of fiber/powder blends may be a way to make dual element fiber reinforced composites. The roll compacting of a particulate mass made up entirely of melt extracted fibers may well be feasible. This would however require a larger quantity of 1 or 2 mm fibers than was possible in this program. Due to their relative softness the fibers behave more like atomized powders than hydrometallurgical powders and are thus more difficult to roll compact. For this reason experience at Sherritt suggests that a substantial experimental program would be necessary to establish a useful roll compacting process and product using melt extracted nickel fibers alone.

6.0 REFERENCES

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Table 1 The Effect of Wheel Speed on Fiber Production
(Sherritt Logbook 1075-4 to 9)

Run No	Wheel Speed		Melt Temp (°C)	Contact Time (s)	Weight Recovered (g)	Average Extraction Rate (g/s)	Wheel Temp(°C)/ at Time (min) After Finish
	(rpm)	(m/s)					
C2	512	4.05	1654	40	47	1.2	88/1
C3	1009	7.98	1656	24	41	1.7	109/~1
C4	1506	11.9	1677	10	31	3.1	113/~3
C5	300	2.37	1651	89	205	2.3	n.d.

Table 2 Fiber Production Using an 18 Notch Wheel
(Sherritt Logbook 1075-10 to 32)

Run No (1)	Wheel Speed		Melt Temp (°C)	Contact Time (s)	Weight Recovered (g)	Average Extraction Rate (g/s)	Wheel Temp(°C)/ at Time (s) After Finish
	(rpm)	(m/s)					
C6	300	2.37	1653	177	400	2.3	346/30
C7	500	3.95	1647	122	120	1.0	162/60
C8	704	5.56	1573	38	84	2.2	Not rec
C9	701	5.54	1648†	122	48	2.5	126/30
C10	701	5.54	1648*	122	46	2.6	125/30

† 30 s after run completed, melt temperature was 1600°C

* 30 s after run completed, melt temperature was 1592°C

(1) All runs used an 18 notch, 90° apex angle wheel (Figure 5)

Table 3 Fiber Production Using Smooth and 54 Notch Wheels
(Sneritt Logbook 1075-32 to 48)

Run No (1)	Melt Temperature (°C)	Contact Time (s)	Weight Recovered (g)	Reason for Termination
C11	1647	37	23	Power kicked out
C12	1656	0	0	Power kicked out
C13	1652	1	<1	Power kicked out
C14	-	-	-	Charge did not melt down
C15	1661	9	2	Power kicked out
C16	1655	28	52	Power kicked out
	1652	37		Freezing in furnace spout

(1) C11 and C12 used a smooth 60° apex angle wheel whilst the others used a 54 notch, 90° apex angle wheel

Part of the product of C11, 13, 15 and 16 was quenched in water

All runs used a wheel speed of 700 rpm (5.53 m/s surface speed)

Table 4 Fiber Production Using 18 and 54 Notch Wheels
(Sherritt Logbook 1075-49 to 55)

Run No (1)	Contact Time (s)	Weight Recovered (g)	Comments
C17	~45	15	Power tripped out
C18	~120	157	No problems. Wheel temperature increased from 27 to 160°C during run
C19	120	240	Higher vertical feed rate
C20	154	90	Power tripped after 34 s, breaker reset and run restarted - terminated voluntarily after a further 2 min
C21	120	99	No problems; fibers quenched in water

- (1) All except C18 used a 54 notch, 90° apex angle wheel. C18 used an 18 notch, 90° apex angle wheel (see Figure 9). All runs used a wheel speed of ~700 rpm (5.5 m/s) and a melt preheat temperature of ~1650°C. Product was collected in a dry tray except for C21.

Table 5 Fiber Production Using 72 and 120 Notch Wheels
(Sherritt Logbook 1075-58 to 67)

Run No (1)	Contact Time (s)	Weight Recovered (g)	Comments
C22	120	32	Wheel temperature increased from 27 to 132°C during run
C23	165	59	Power trip after 30 s, then after a further 15 s then no problem for 120 s
C24	240	100	Power trip after 10 s, then after a further 120 s. Furnace recharged then ran 110 s before another power trip
C25	75	6	Power trip after 75 s, could not restart due to spout metal freeze up
C26	75	3	Power trip after 30 s then again after a further 45 s.
C27	225	31	Ran 120 s, cooled the wheel then ran again

(1) C22, 23 and 24 used a 72 notch, 90° apex angle wheel. C25, 26 and 27 used a 120 notch, 60° apex angle wheel (Figure 10). All runs used a wheel speed of 700 rpm (5.5 m/s surface speed) and a melt preheat temperature of 1650 to 1675°C

Table 6 Fiber Production Using a 72 Notch Wheel
(Sherritt Logbook 1075-68 to 70)

Run No (1)	Contact Time (s)	Weight Recovered (g)	Comments
C28	90	13	Power tripped out
C29	360	73	Three 120 s runs with time for wheel cooling in between; no power trips
C30	320	84	Two 120 s runs followed by an 80 s run concluded due to melt freeze up. Pauses in between to allow wheel to cool; no power trip

- (1) All runs used a 72 notch, 90° apex angle wheel. Wheel speed was 700 rpm (5.5 m/s surface speed) and the nickel melt preheat temperature varied in the range 1660 to 1680°C

Table 7 Oxygen Contents of Melt Extracted Fibers, Water Atomized Powders and Hydrometallurgical Powders
(Sherritt Logbook 1075-77)

Sample Identification	O ₂ Content (1) (%)
Hydrometallurgical	
BRS 4757	0.15
BRS 4757 (repeat test)(2)	0.15
Water Atomized	
MA 7364	0.04
MA 7364 (repeat test)(2)	0.04
MA 7117	0.06
Melt Extracted (3)	
C19	0.15
C20	0.14
C21	0.19
Blend of C22, 23, 24	0.20
Blend of C28, 29, 30	0.25
Blend of C25, 26, 27	0.21

- (1) Determined according to guidelines set out in MPIF Standard 02-1977 Revision
- (2) According to the Standard, duplicate results within $\pm 0.01\%$ are acceptable
- (3) C21 was quenched in water and the rest were air cooled. C19 to 21 were produced from the 54 notch wheel; C22 to 24 and 28 to 30 from the 72 notch wheel; C25 to 27 from the 120 notch wheel

Table 8 Hardness of Melt Extracted, Atomized and Hydrometallurgical Particles

Nickel Particle Sample	Hardness VHN (1)	
	Mean (2)	Standard Deviation
Melt Extracted Run C20 (Air Cooled)	111	15
Melt Extracted Run C21 (Water Quenched)	141	19
Water Atomized Run MA 7117	93	16
Hydrometallurgical Batch BRS-4757	208	11

- (1) Each number represents ten individual hardness determinations, mostly on different particles. The load applied was 100 g
- (2) C20 and C21 are not significantly different at the 99% statistical confidence level

Table 9 Transverse Rupture Strength of Melt Extracted, Water Atomized and Hydrometallurgical Nickel Powder Compacts

Sample (1)	Weight (g)	Compacting Pressure (MPa)	Test Piece Thickness (mm)	Compact Density (%) (2)	Rupture Load (kg)	TRS (3) (MPa)	No of Tests Averaged
Melt Extracted							
C19	15.97	418	5.12	86.8	54.9	61.7	2
C20	16.02	418	5.26	84.6	29.9	31.8	2
C21	16.00	418	5.31	83.8	31.1	32.4	2
Blend of C22, 23 and 24	15.96	418	5.22	85.0	23.1	24.9	2
Blend of C25, 26 and 27	15.96	418	5.32	83.4	6.5	6.8	1
Water Atomized							
Heat MA 7117	15.99	418	5.51	80.7	3.2	3.2	2
	18.32	418	6.21	82.1	6.2	4.7	7
	18.58	506	6.01	85.2	10.1	8.1	3
Heat MA 7364	15.94	418	5.28	84.0	6.8	7.2	2
	18.54	418	6.09	84.7	9.2	7.3	2
	18.56	506	5.95	86.7	10.6	8.8	2
Hydrometallurgical							
Batch BRS 1827	15.98	418	6.28	70.8	15.6	11.6	3
Batch BRS 4757	15.95	418	6.28	70.6	16.0	11.9	3

(1) Powder compacts were prepared and tested according to guidelines set out in MPIF Standard 15 1985 Revision, except that thickness varied outside the nominal recommended range of 6.32 to 6.38 mm

(2) Shown as percent of theoretical solid density (8.9 g/cm³)

(3) Given by $\frac{3 \times (\text{Rupture Load}) \times (\text{Testpiece Span}) \times 9.81}{2 \times (\text{Testpiece Thickness})^2 \times (\text{Testpiece Width})}$ (Pa)

Where Testpiece span = distance between supports = 25.4 mm, and testpiece width = 12.7 mm. All linear dimensions in the equation are expressed in m

Table 10 Tensile Strength of Melt Extracted, Water Atomized and Hydrometallurgical Nickel Powder Compacts

Sample (1)	Weight (g)	Compacting Pressure (MPa)	Test Piece Thickness (mm)	Compact Density (%) (2)	Rupture Load (kg)	Tensile Strength (MPa)(3)	No of Tests Averaged
Melt Extracted							
C19	14.62	413	3.00	85.0	14.7	8.5	2
C20	14.56	413	3.02	84.0	14.8	8.5	2
C21	14.58	413	3.08	82.5	9.7	5.5	2
Blend of C22, 23 and 24	14.55	413	3.00	84.6	11.4	6.6	2
Blend of C25, 26 and 27	14.54	413	3.18	79.8	1.5	0.8	1
Water Atomized							
Heat MA 7117	14.48	413	3.06	82.4	1.0	0.6	1
	23.89	413	5.45	76.4	1.0	0.3	2
	28.29	413	6.41	76.7	1.6	0.4	3
Heat MA 7364	14.51	413	3.03	83.4	1.8	1.0	2
	23.87	413	5.36	77.5	1.3	0.4	2
	28.31	413	6.36	77.5	1.3	0.4	3
Hydrometallurgical							
Batch BRS 4757	14.50	413	3.32	76.1	7.6	4.0	2
	23.80	413	6.48	64.2	5.4	1.4	2

(1) Powder compacts were prepared and tested according to guidelines set out in MPIF Standard 10 1963 Revision, except that thickness varied outside the nominal recommended range of 5.08 to 6.35 mm

(2) Shown as percent of theoretical solid density (8.9 g/cm³)

(3) Given by
$$\frac{(\text{Rupture Load}) \times 9.81}{\text{Thickness} \times \text{Gauge Width}} \quad (\text{Pa})$$

Where gauge width = 5.67 mm and all linear dimensions in the equation are expressed in m

Table 11 Roll Compacting of Melt Extracted Fibers, Water Atomized Powders and Hydrometallurgical Powders
(Sherritt Logbook 1075-71)

Nickel Powder/Fiber Blend (1)	Roll Compacted Strip Dimensions		Test No
	Thickness (mm)	Density (%) (2)	
100% Water Atomized MA 7117	0.88	76.1	1
95% MA 7117, 5% Blend of C22, 23, 24	0.94	79.9	2
100% Water Atomized MA 7364	0.97	84.3	3
95% MA 7364, 5% Blend of C22, 23, 24	1.05	87.1	4
75% MA 7364, 25% Blend of C22, 23, 24	1.35	91.5	5
100% Hydrometallurgical BRS 4757	0.76	55.3	6
95% BRS 4757, 5% Blend of C28, 29, 30	0.92	70.4	7
75% BRS 4757, 25% Blend of C28, 29, 30	1.14	79.8	8
65% BRS 4757, 25% Blend of C28, 29, 30, 10% MA 7364	1.21	87.9	9

- (1) All extracted fibers were made using the 74 notch wheel and had a length of ~2 mm
- (2) Based upon a theoretical solid density of 8.9 g/cm³

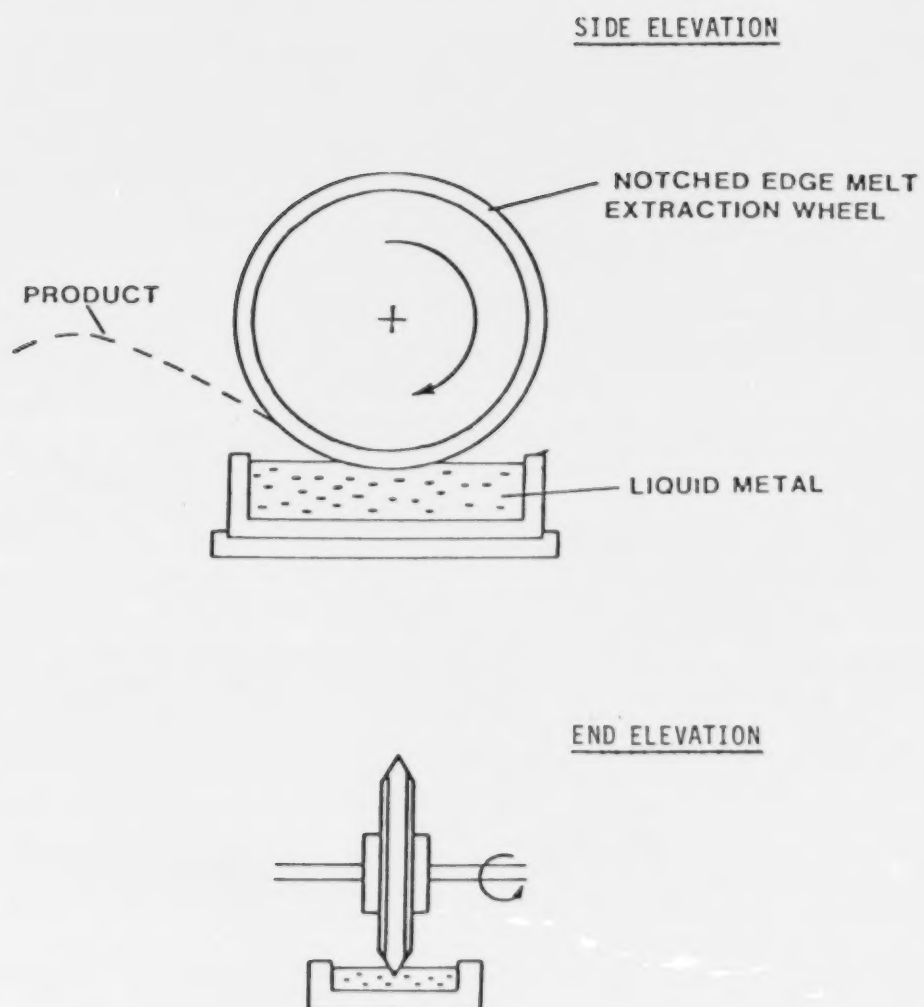


Figure 1 Schematic Illustrating Crucible Melt-Extraction
Mode of Rapid Solidification

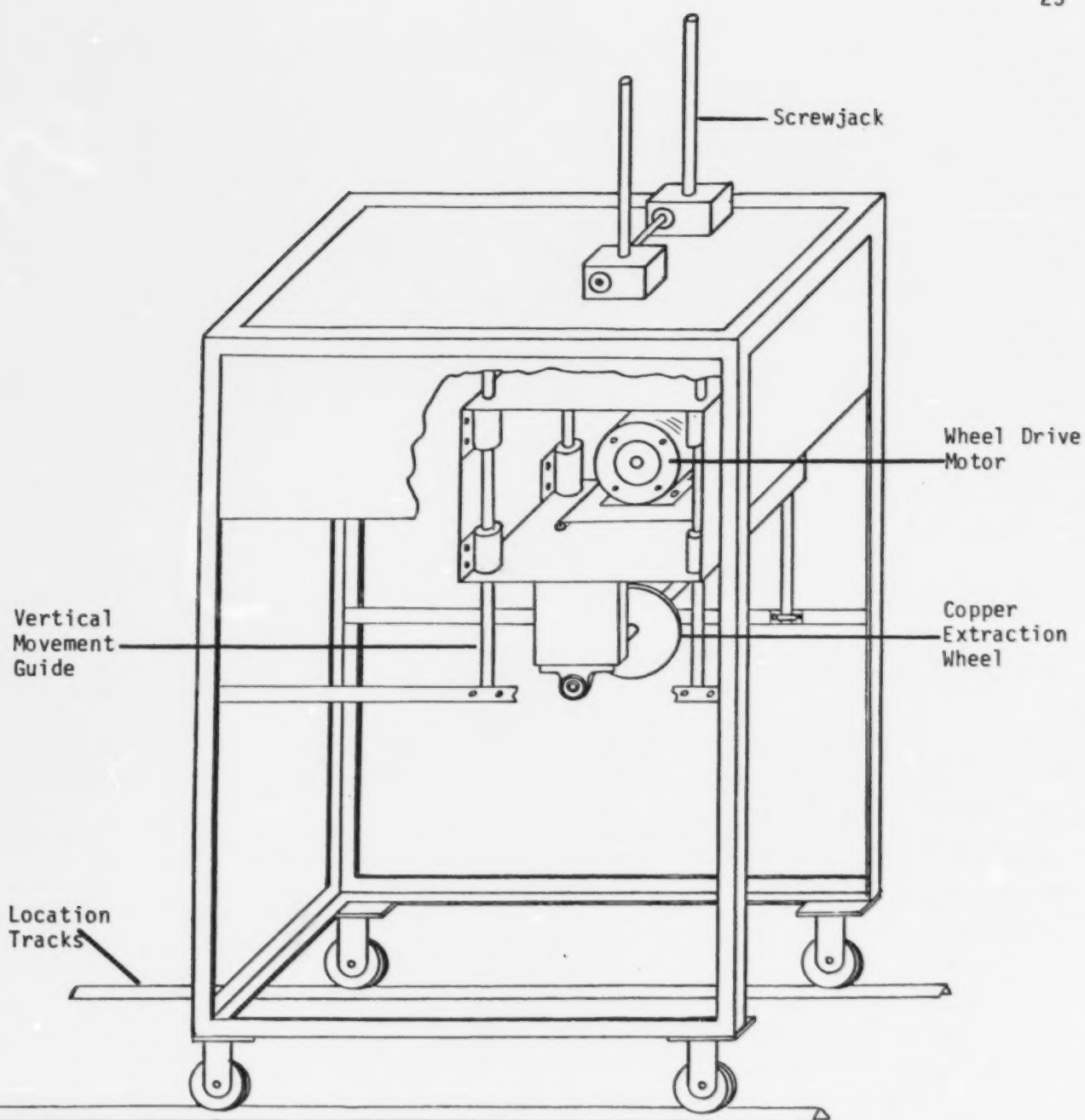


Figure 2 Melt-Extraction Assembly

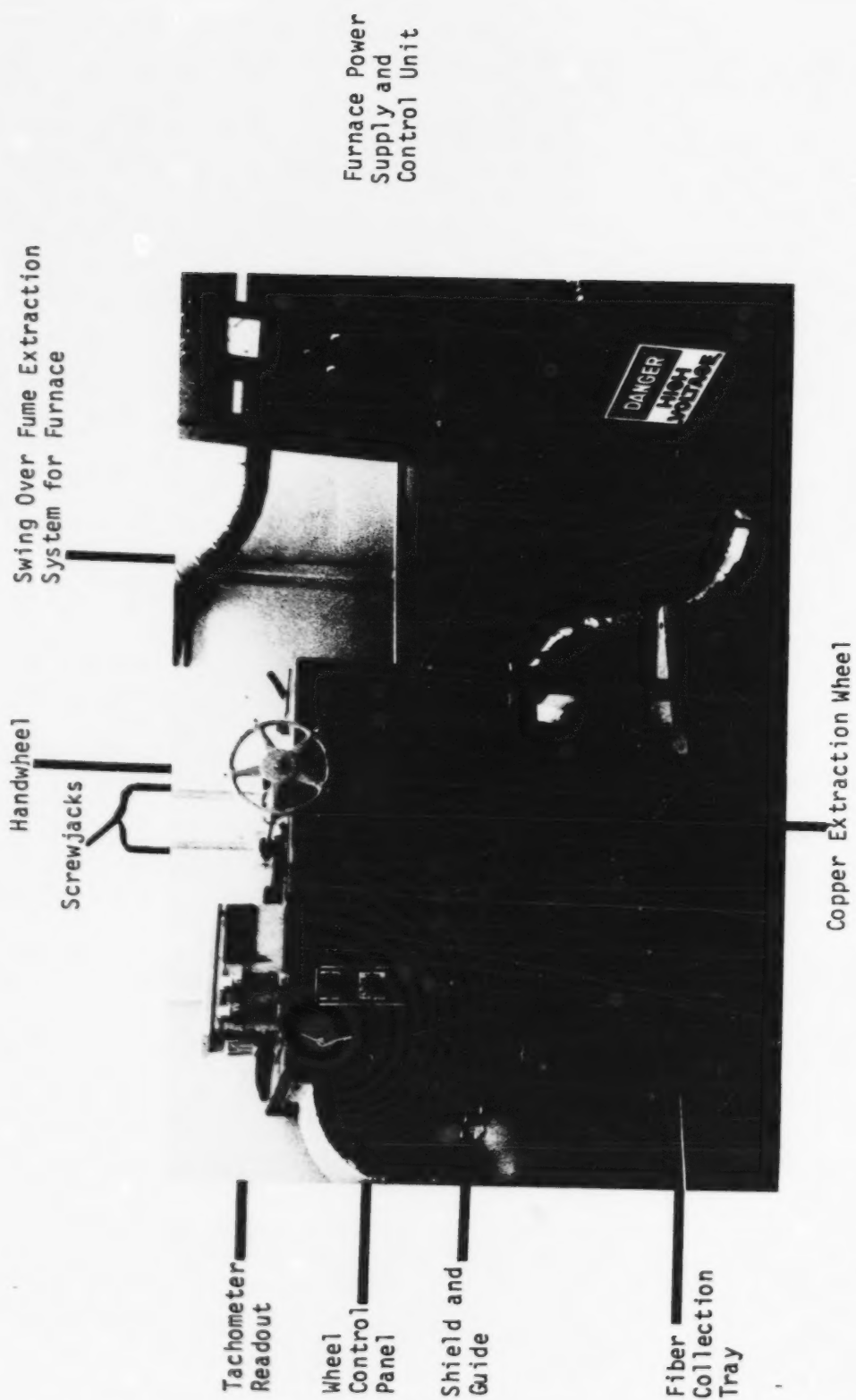
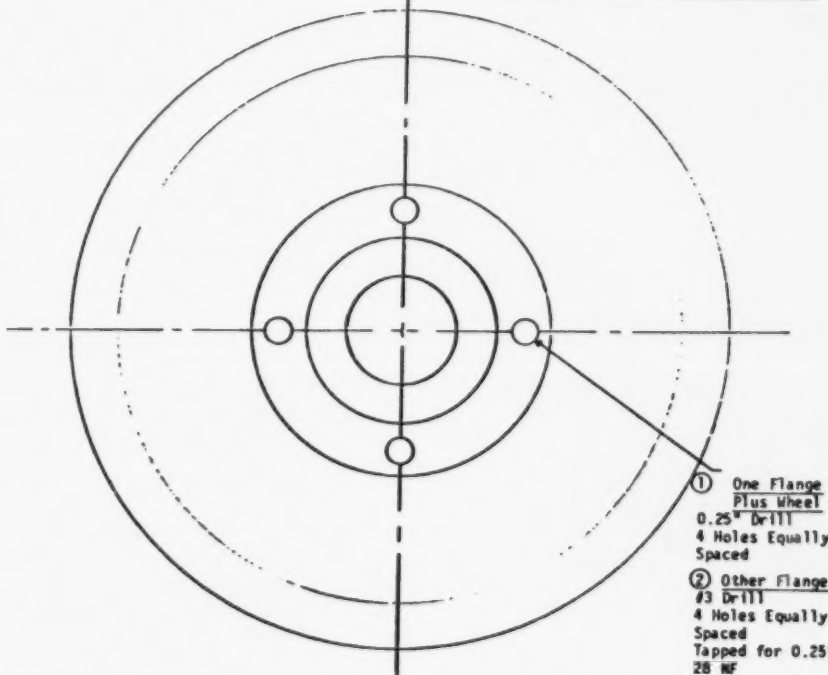
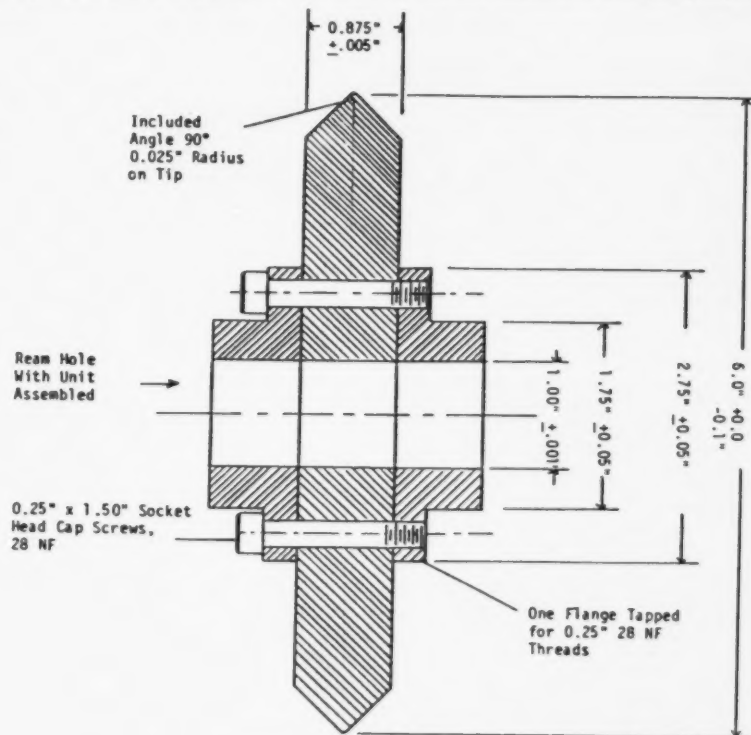


Figure 3 Melt-Extraction Apparatus

Figure 4

Sketch Pad

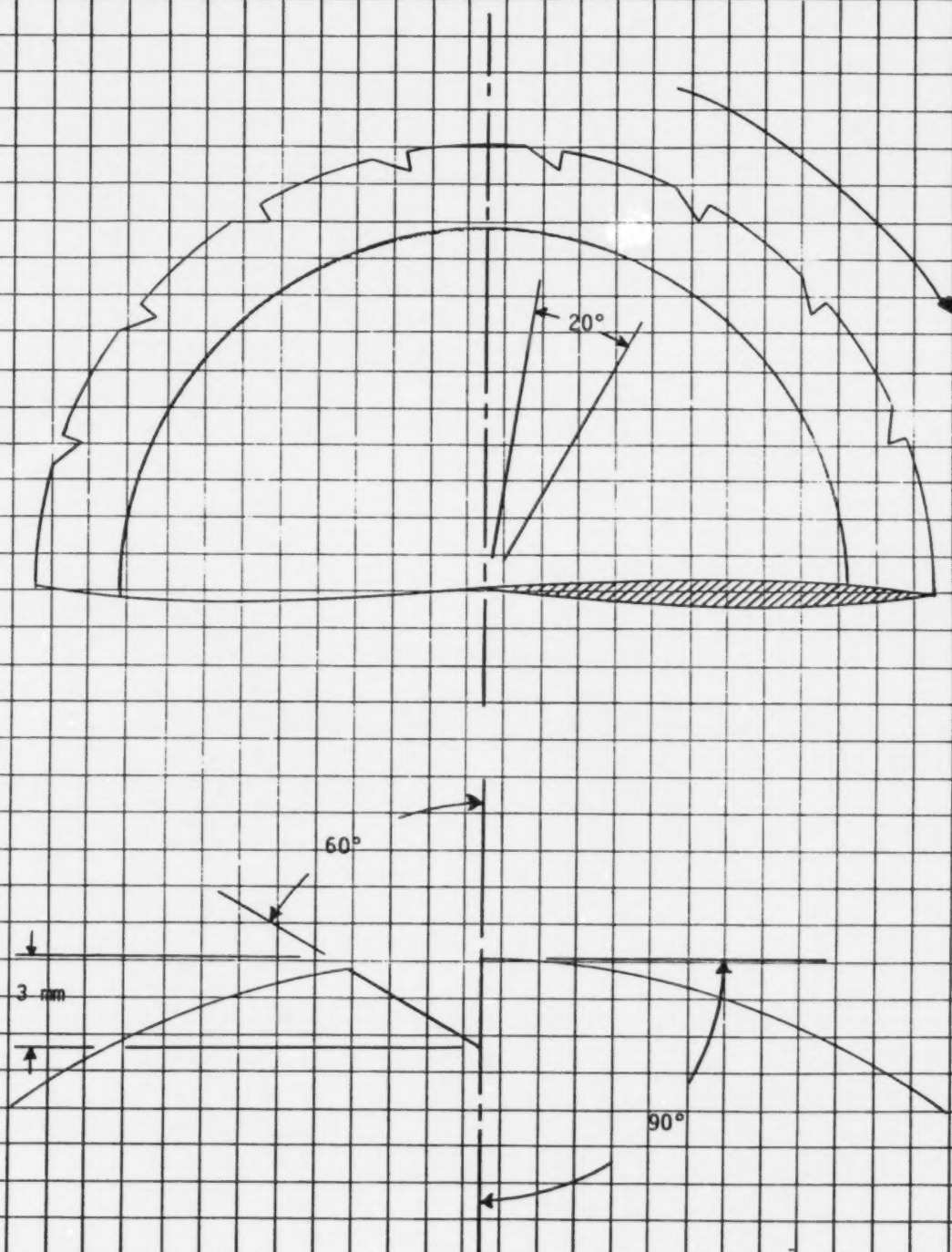


TITLE MELT EXTRACTION WHEEL AND FLANGE		LOCATION RESEARCH	
DRAWN BY J. TAMELING	CHECKED BY M. RUSCOE	P.E. No.	N/A
PROJECT No. 5702	DATE APRIL 17, 1985	SKETCH No.	1

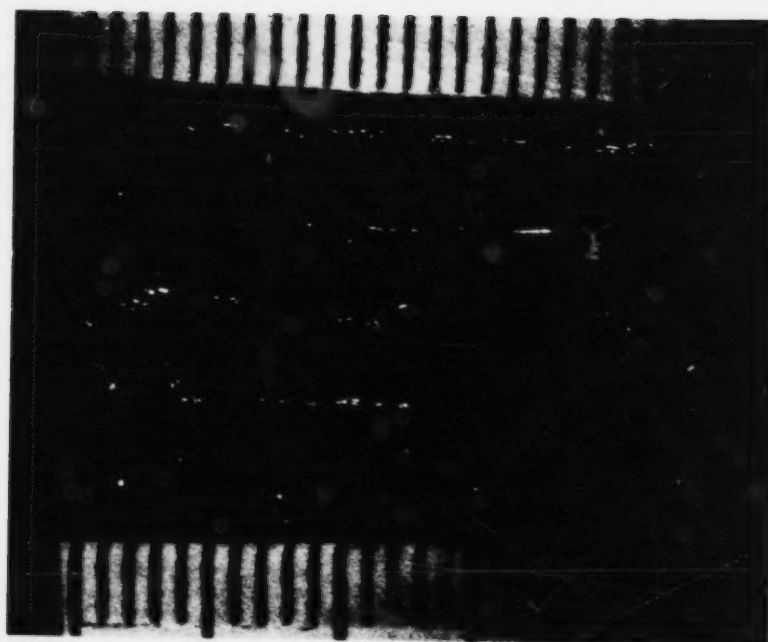
sherritt

Figure 5

Sketch Pad

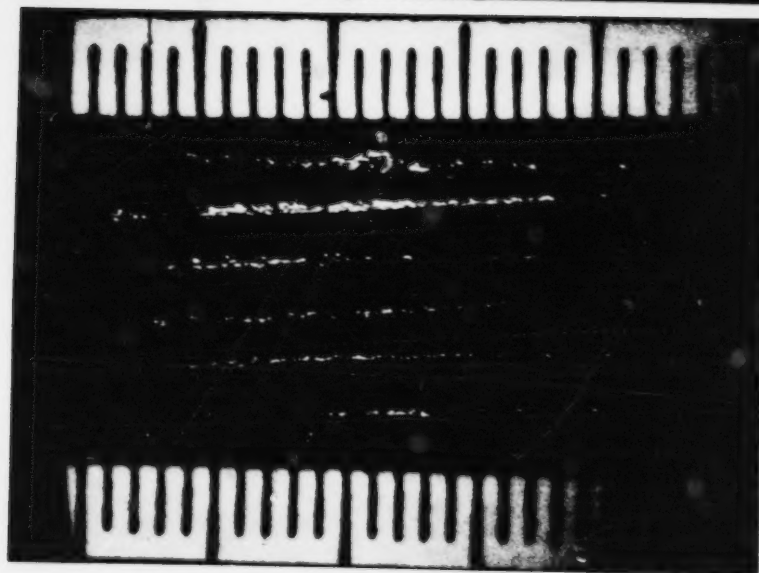


TITLE MELT EXTRACTION WHEEL 2		LOCATION RESEARCH
DRAWN BY J. TAMELING	CHECKED BY <i>MMR</i>	P.E. No. N/A
PROJECT No. 5702	DATE JUNE 28, 1985	SKETCH No. 2



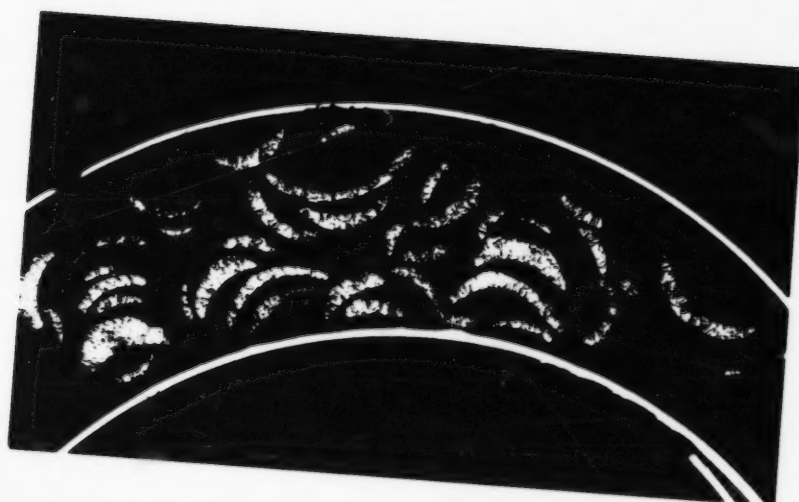
Run C6

1 scale
division
= 1 mm



Run C7

Figure 6 Melt-Extracted Nickel Fiber Shapes (18 Notch Wheel)
(X4)



Run C8
(Mold 5141)

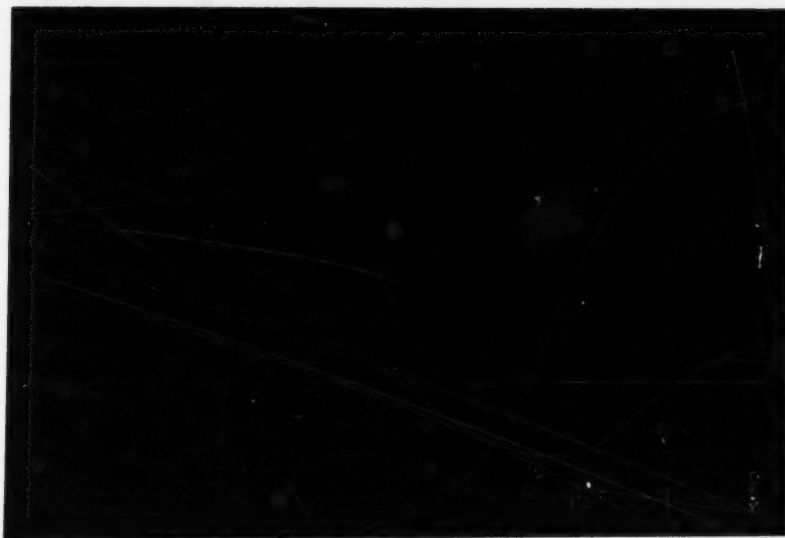
Figure 7 Transverse Section of Melt-Extracted Nickel Fibers
(X20)



A
Melt-Extraction
Run C8
(Mold 5141)



B
Melt-Extraction
Run C8
(Mold 5141)



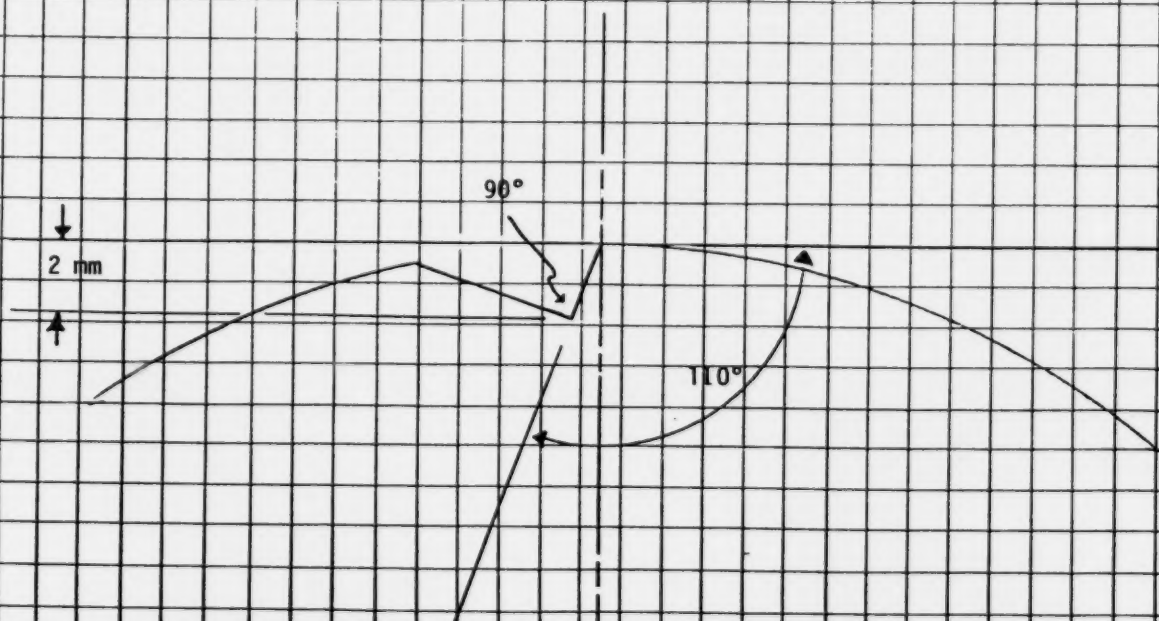
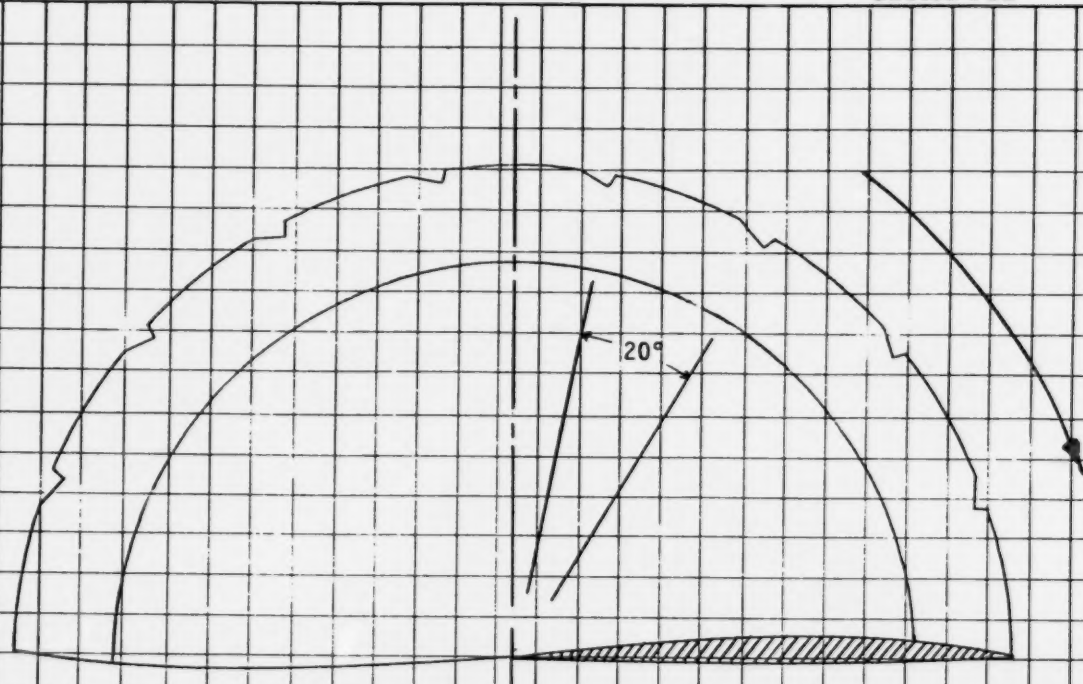
C
Atomizing
Run MA 7117
(Mold 5073)

Figure 8 Microstructures of Melt-Extracted Nickel Fibers
and a Water Atomized Nickel Particle (X500)

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Figure 9

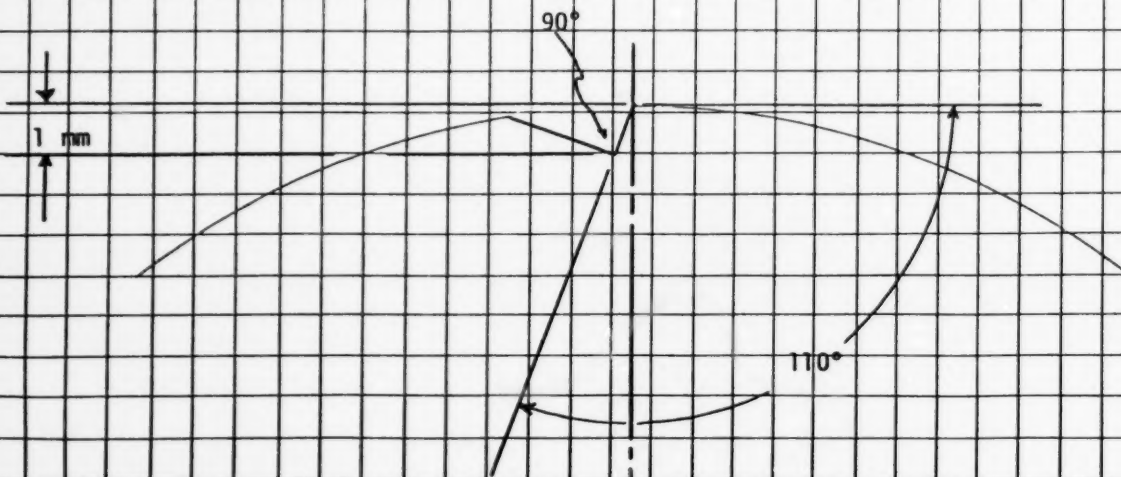
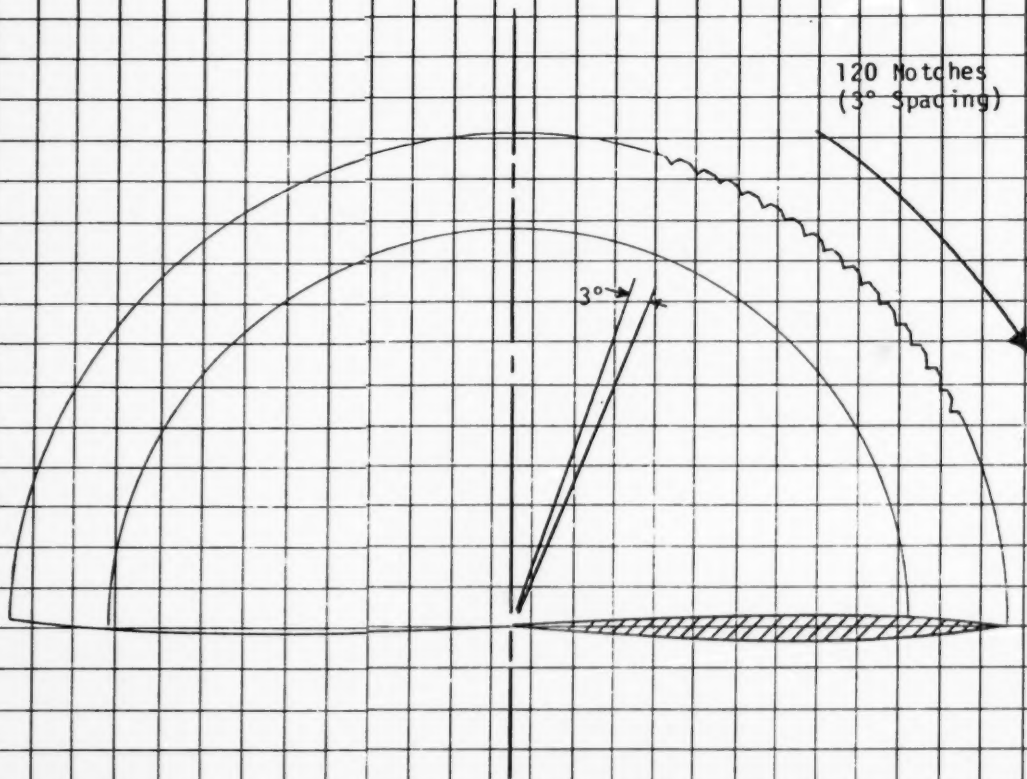
Sketch Pad



TITLE MELT EXTRACTION WHEEL 1 (modified)		LOCATION RESEARCH
DRAWN BY J. TAMELING	CHECKED BY	P.E. No. N/A
PROJECT No. 5702	DATE JUNE 20, 1985	SKETCH No. 4

sherritt

Sketch Pad



TITLE MELT EXTRACTION WHEEL #3 (60° APEX ANGLE)

LOCATION RESEARCH

DRAWN BY J. TAMELING

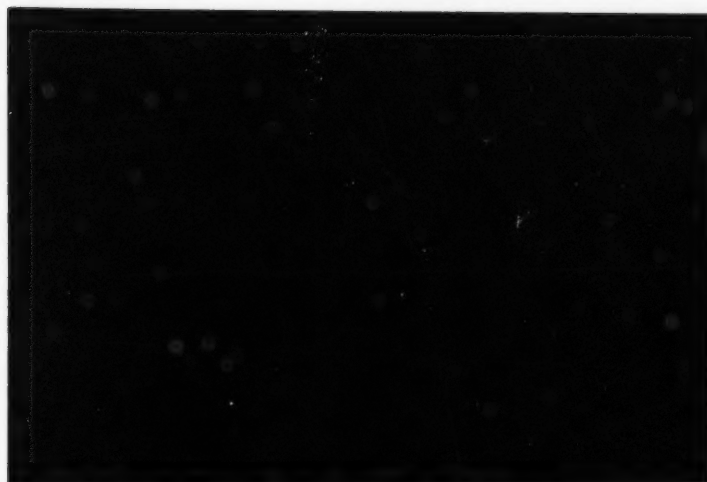
CHECKED BY

P.E. No. N/A

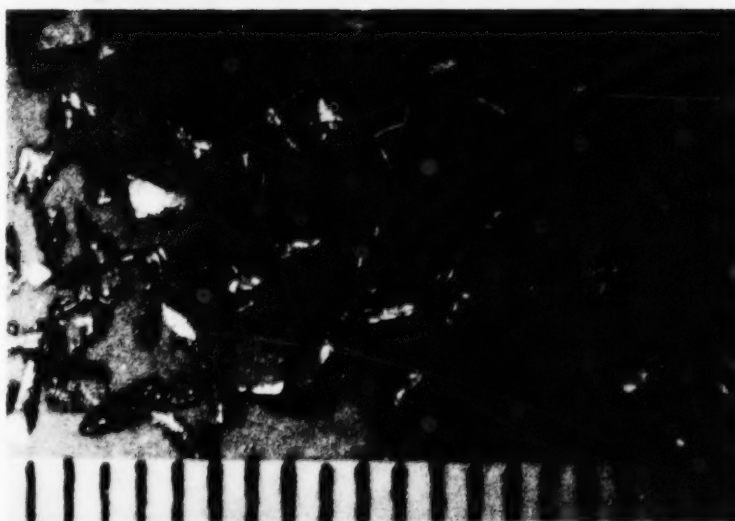
PROJECT No. 5702

DATE AUGUST 14, 1985

SKETCH No. 6



C13
(54 Notch Wheel)
(X10)

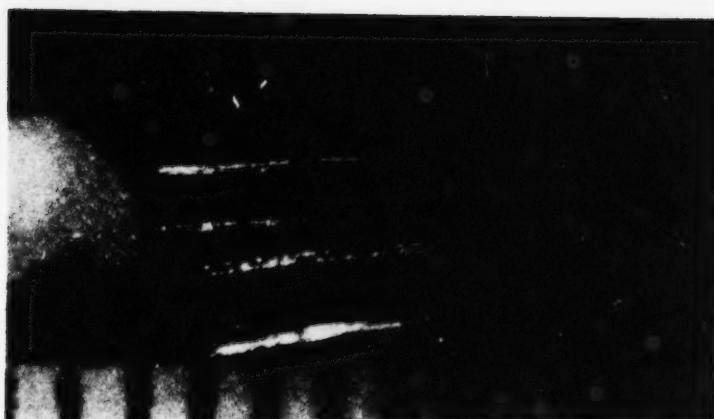


C23
(72 Notch Wheel)
(X5)

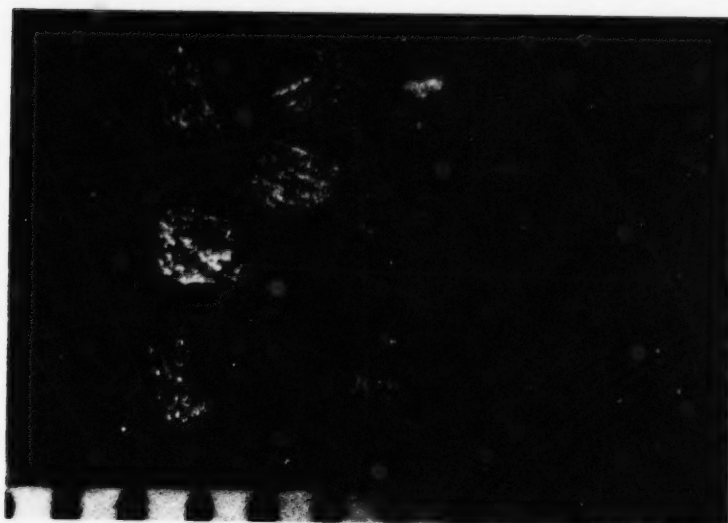


C25
(120 Notch Wheel)
(X30)

Figure 11 Sherritt Melt-Extracted Nickel Fibers

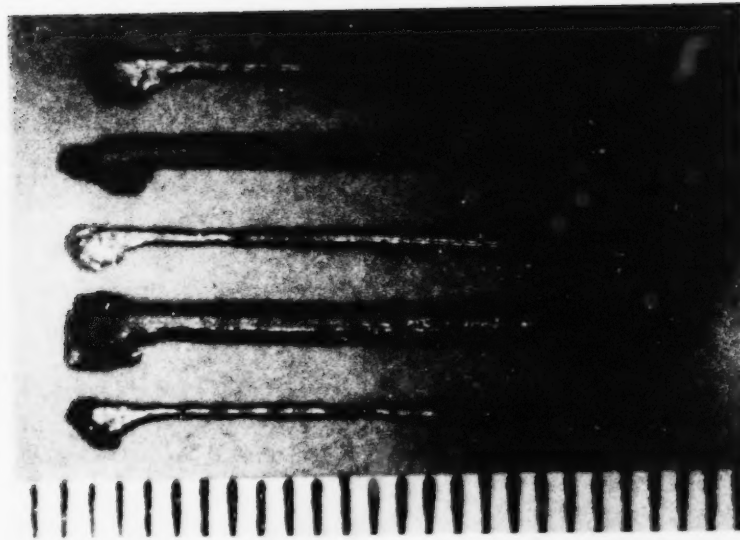


Melt-Extraction

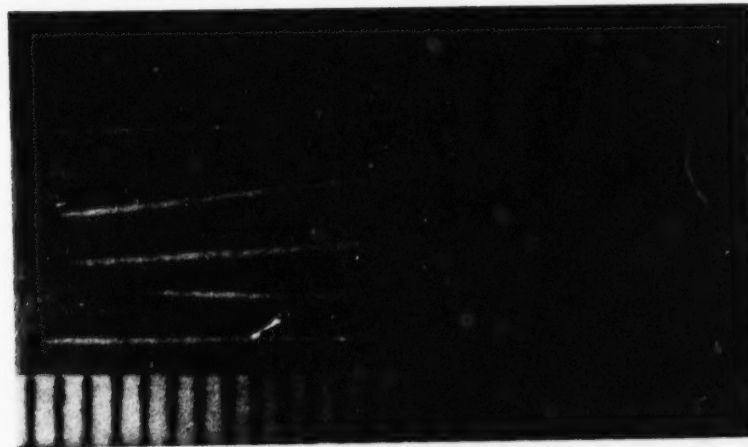


Melt-Spinning

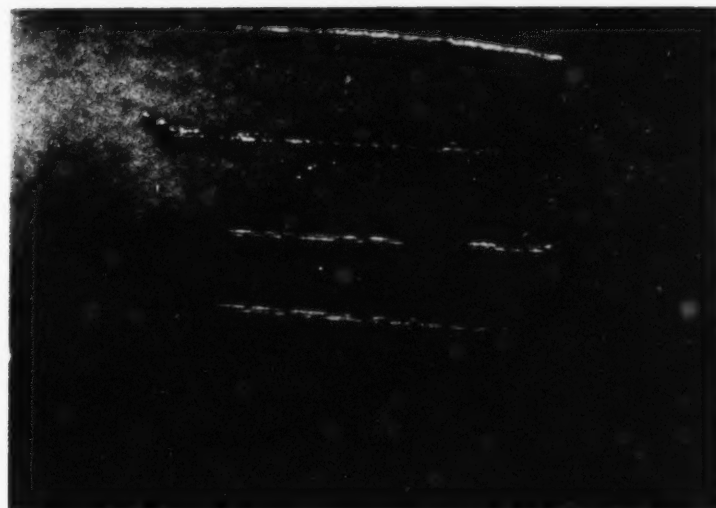
Figure 12 Aluminum Particulates from Transmet
(X10)



Melt-Extraction
Carbon Steel
(X4)



Melt-Extraction
Type 304
Stainless Steel
(X4)



Melt Overflow
(X10)

Figure 13 Particulates from Ribtec



Melt-Extraction

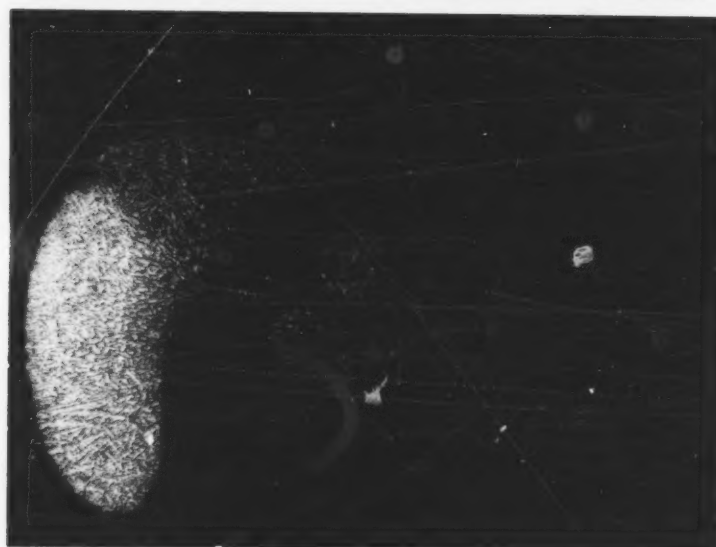


Melt-Spinning

Figure 14 Aluminum Particulates from Trānsmet
Transverse Cross Sections
(Mold 5170, X100)



Melt-Extraction
Carbon Steel

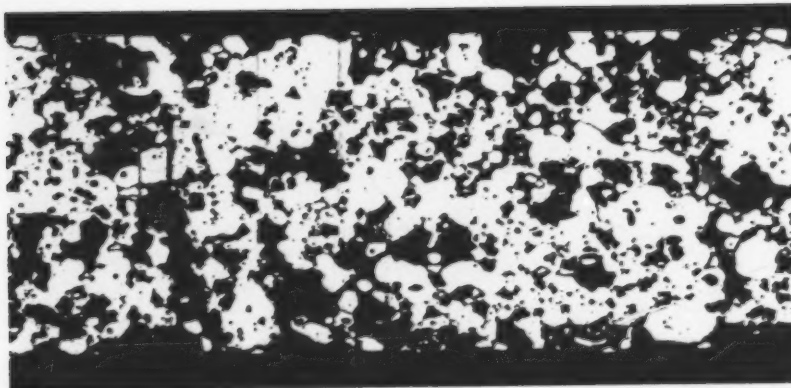


Melt-Extraction
Type 304
Stainless Steel

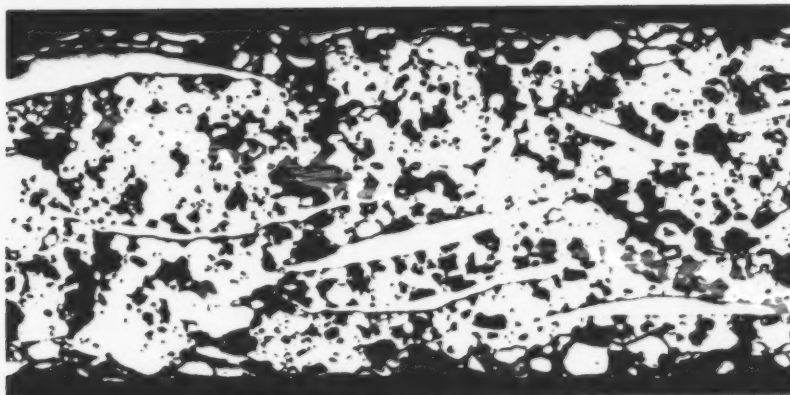


Melt Overflow

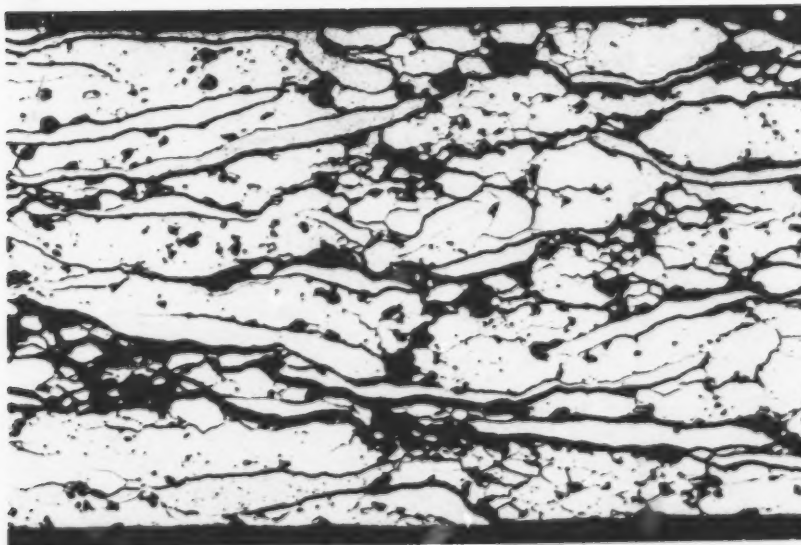
Figure 15 Particulates from Ribtec
Transverse Cross Sections
(Mold 5170, X100)



Test 3
MA 7364

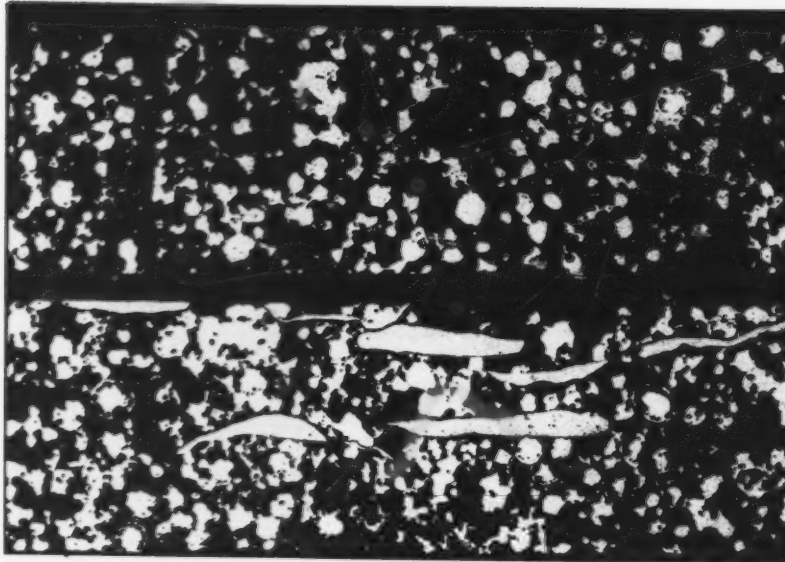


Test 4
95% MA 7364
5% Blend of
C22, 23, 24

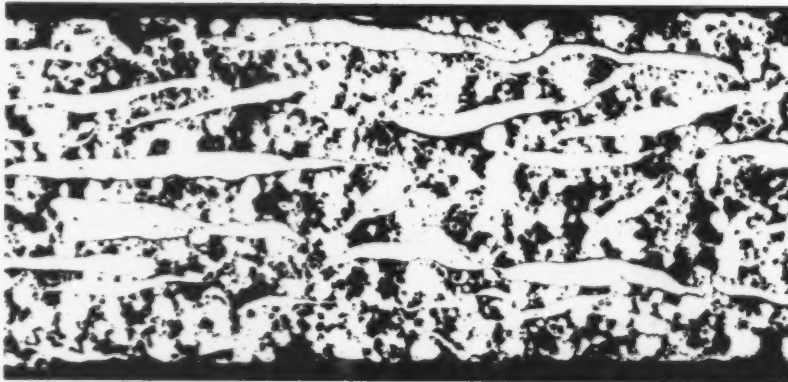


Test 5
75% MA 7364
25% Blend of
C22, 23, 24

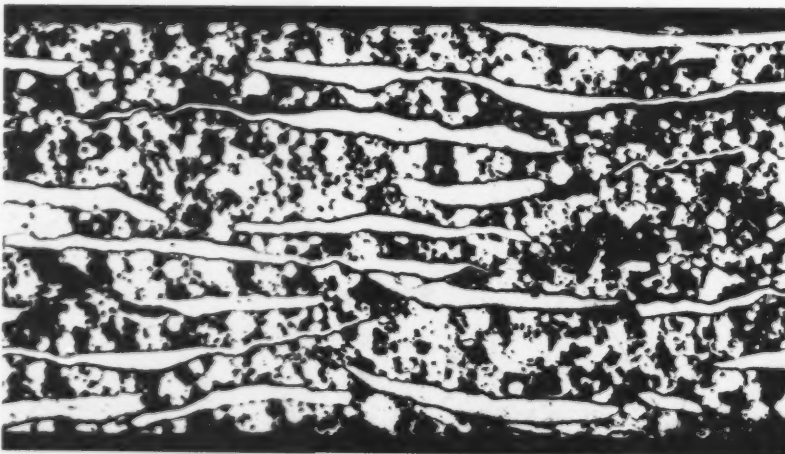
Figure 16 Roll Compacted Strip Based on MA 7364
Transverse Cross Sections
(Mold 5762, X50)



Test 6
BRS 4757



Test 7
95% BRS 4757
5% Blend of
C28, 29, 30



Test 9
65% BRS 4757
25% Blend of
C28, 29, 30
10% MA 7364

Figure 17 Roll Compacted Strip Based on BRS 4757
Transverse Cross Sections
(Mold 5762, X50)

